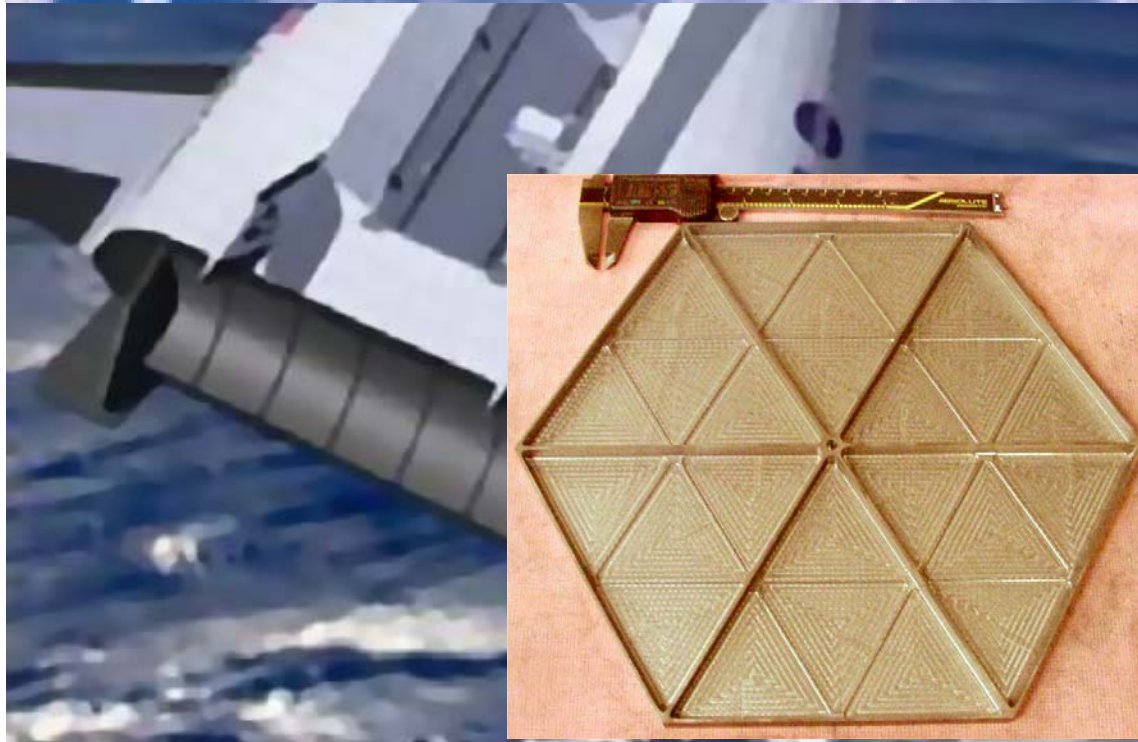




Development of Graphite Fiber Reinforced Mg Alloys for Use as Space and Tactical Mirrors

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Metal Matrix Cast Composites, LLC



Presented to: Technology Days in the Government Mirror Development and Related Technologies Review

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Presentation Outline

- ◆ **Acknowledgements**
- ◆ **Phase I objectives**
 - Desired mirror properties
- ◆ **Constitutive materials properties**
 - Theoretical calculations
- ◆ **Manufacturing approach**
 - Fiber architecture
 - Preform processing
 - Pressure infiltration casting
- ◆ **Program experimental results**
 - Metallography
 - CTE Calibration Curves
 - Demonstration of linear CTE with temperature
 - Comparison of Mg/Gr to other mirror materials
- ◆ **Initial thermal seasoning studies**
- ◆ **Demonstration of machinability**
- ◆ **Si deposition and polishing**
- ◆ **Manufacturing scale-up potential**
- ◆ **Summary of results**
- ◆ **Phase II Objectives, Work Plan and results**
- ◆ **Phase I Replication membrane**
- ◆ **Phase I EKV primary mirror**

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- Dr. Lawrence E. Matson, AFRL/MLLN
- Dr. David Mollenhauer, AFRL/MLLN

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F33615-03-C-5208: “II Low Cost Lightweight Graphite Fiber Reinforced Mg Alloys for Space & ABL Mirrors” and the following:

- Dr. Kevin Kendig, AF Project Manager
- Dr. Lawrence E. Matson, AFRL/MLLN
- Dr. Phil Stahl, NMSEC

Mg/Gr Mirrors: Phase I Technical Objectives

- Produce a dimensionally stable metal matrix composite with a CTE less than 2 ppm/K
- Achieve an areal density of $\sim 5 \text{ kg/m}^2$
- Produce a thermally stable composite with a thermal conductivity of $>150 \text{ W/mK}$
- Demonstrate optical surface finish at $\sim \lambda/200$ to $\lambda/500$
- Investigate preform technologies for achieving isotropic properties
- Demonstrate low cost manufacturing

Desirable Mirror Substrate Properties

- ◆ High stiffness
 - ◆ Low density
 - ◆ High thermal conductivity
 - ◆ Low thermal expansion
 - ◆ Low Cost
 - Low basic materials cost
 - Low processing cost
 - Short lead time for delivery of pre-figured blanks
 - Short polishing times
- Leads to high specific stiffness- E/ρ
- Leads to high thermal stability- k/α

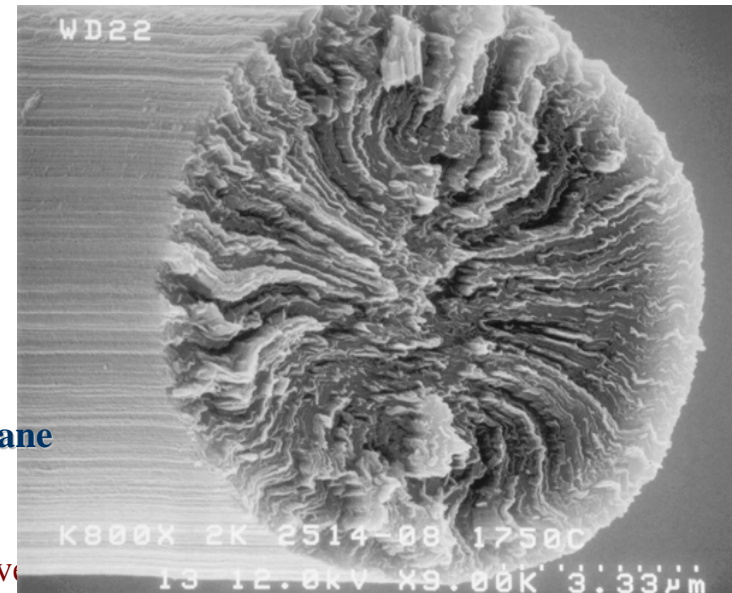
Constituent Materials

Matrix Alloys	Composition	E (gPa)	CTE (ppm/K)	TC (W/mK)
Mg- AZ-91D	Mg- 9 Al- .13 Mn- 1 Zn	45 (6.5 msi)	26	72
Mg- AZ-31	Mg- 3 Al- 0.2 Mn- 1 Zn	45	“ 26	96
Mg- ZC-61	Mg- 6 Zn- 3 Cu- 0.5 Mn	45	“ 26	122
Al 413-HP	Al-12.5 Si- 0.3Mg	71	“ 21	167

Graphite Fiber Reinforcement

	<u>E₁ gPa (msi)</u>	<u>E₂ gPa (msi)</u>	<u>CTE ppm/K</u>
*CKD-x: 862 (125)	6.9	(1)	-1.5
*CKA-x: 965 (140)	6.9	(1)	-1.5

*Cytec graphite fibers: 1” chopped, processed into a random, in plane mat



MMCC's Approach to Mirror Substrate Materials Design and Processing

- **Select Mg reinforced with high modulus graphite fibers**
- **Design preform**
 - **Planar isotropic with discontinuous fibers (Ph. I)**
 - **Quasi-isotropic preform with continuous fibers (Ph. II)**
 - **Hybrid of discontinuous P-I and continuous Q-I preform (Ph. II)**
 - **Organize architecture so that planar-isotropic preforms coincide with reflective plane of mirror**

MMCC's Approach to Mirror Substrate Materials Design and Processing- (continued)

- **Compress preform to volume fraction required for target coefficient of thermal expansion (CTE). Preform to be pressed to near final figure.**
- **Preform loaded into a near final figure machined (carbon) mold and vacuum/pressure infiltrated with molten Mg or Al alloy**
- **Infiltrated blank lightweight machined on back face, figure machined and Si coated prior to polishing to final figure and finish**

Modified Schapery's Equation for Composite CTE Prediction

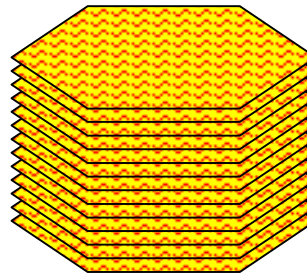
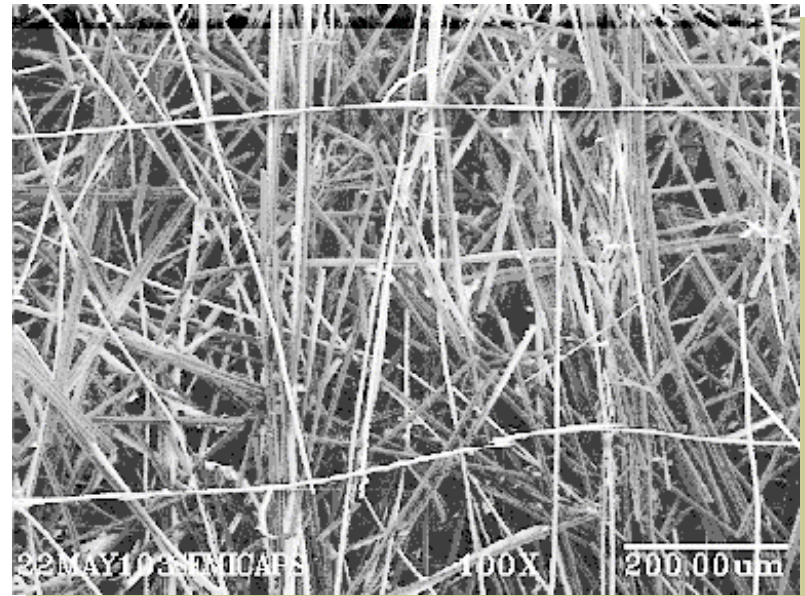
$$\alpha_{cx} = \frac{\alpha_m E_m v_m + \alpha_{fa} E_{fa} \frac{v_f}{2} + \alpha_{ft} E_{ft} \frac{v_f}{2}}{E_m v_m + E_{fa} \frac{v_f}{2} + E_{ft} \frac{v_{ft}}{2}}$$

Where:

α_{cx} = coefficient of thermal expansion of composite
 E = Young's modulus of elasticity
 v = volume fraction reinforcement

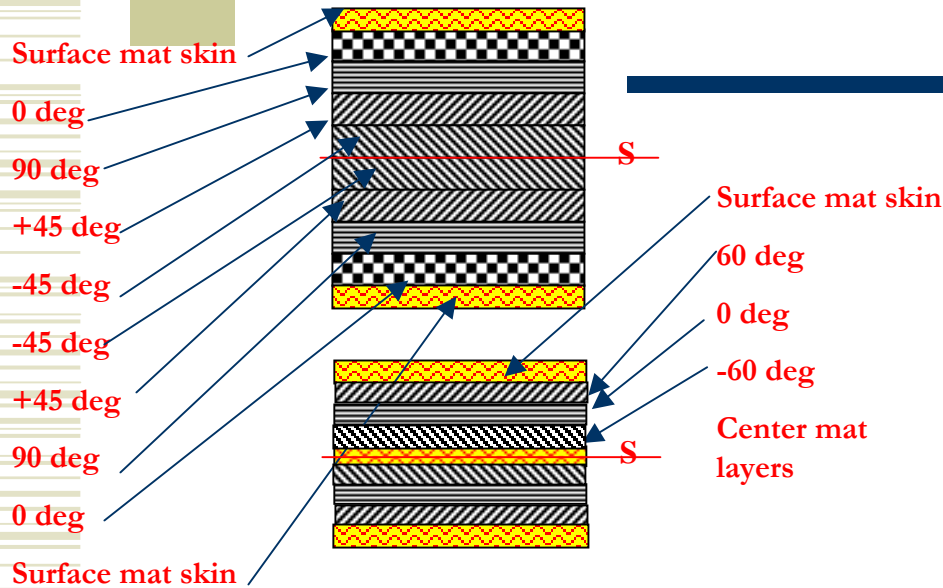
Subscripts:

m = matrix
 f = fiber
 fa = fiber axial
 ft = fiber transverse



Planar-isotropic architecture results from discontinuous fibers randomly distributed in-plane

Concepts for Continuous/Discontinuous Hybridization of Planar Isotropic Preforms

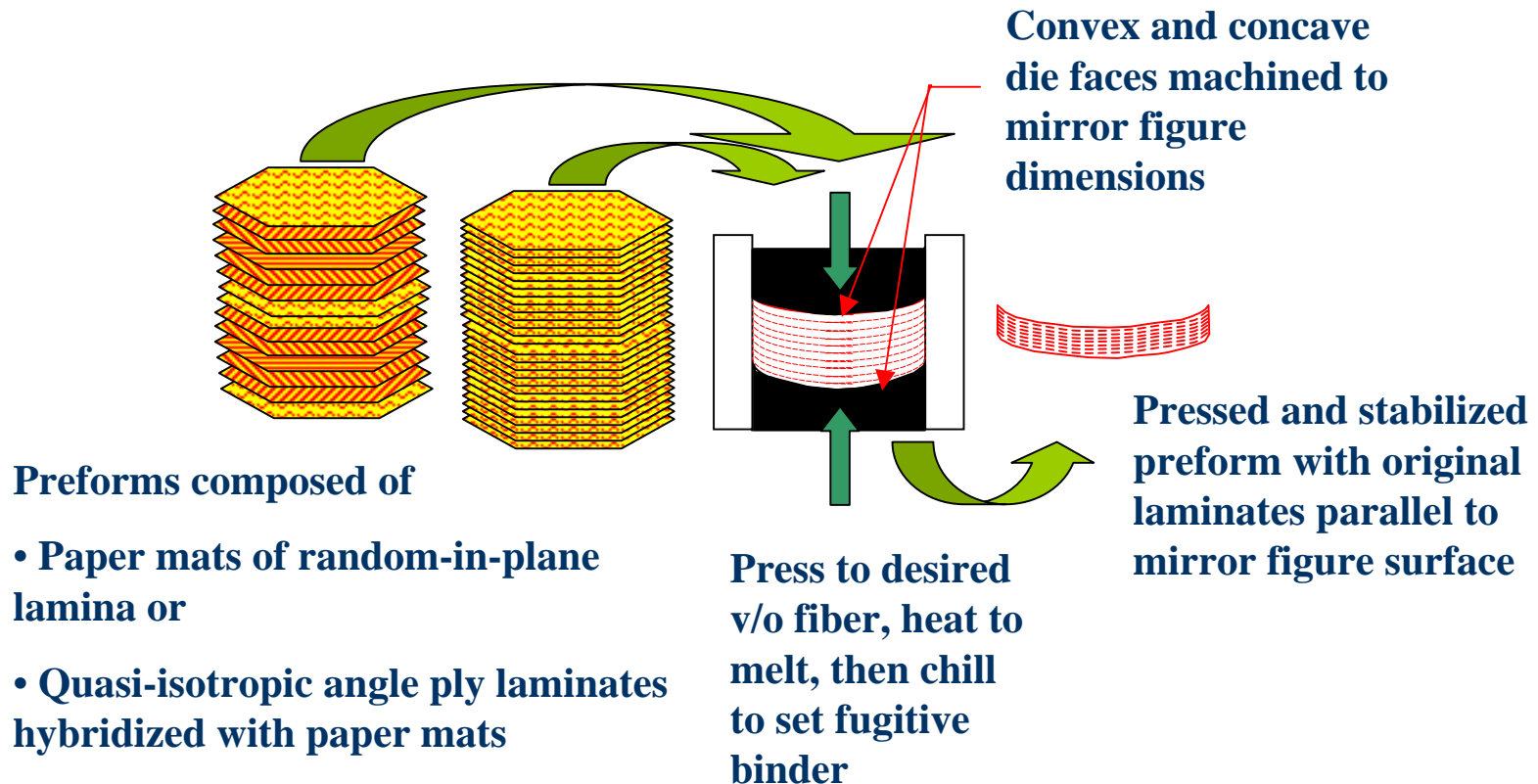


- Hybridized skin
- Quasi-isotropic symmetrical angle-ply laminates forming substrate
- Back skin to provide balance and actuator attachment surface
- Ni or Si plated/deposited surface

$(0^\circ + 60^\circ - 60^\circ / -60^\circ + 60^\circ 0^\circ) \times n$
+ mat skin + inner layers

Section of base structure. Surface skin is co-infiltrated graphite veil or Saffil™ paper to prevent fiber print-through and to provide a surface for machining/polishing.

Discontinuous or Continuous Angle Ply Graphite, (or Hybridized) Fiber Preform Manufacture



Manufacturing Mg/Gr Mirror Substrates at MMCC

Preforms with original preform laminates parallel to mirror surface

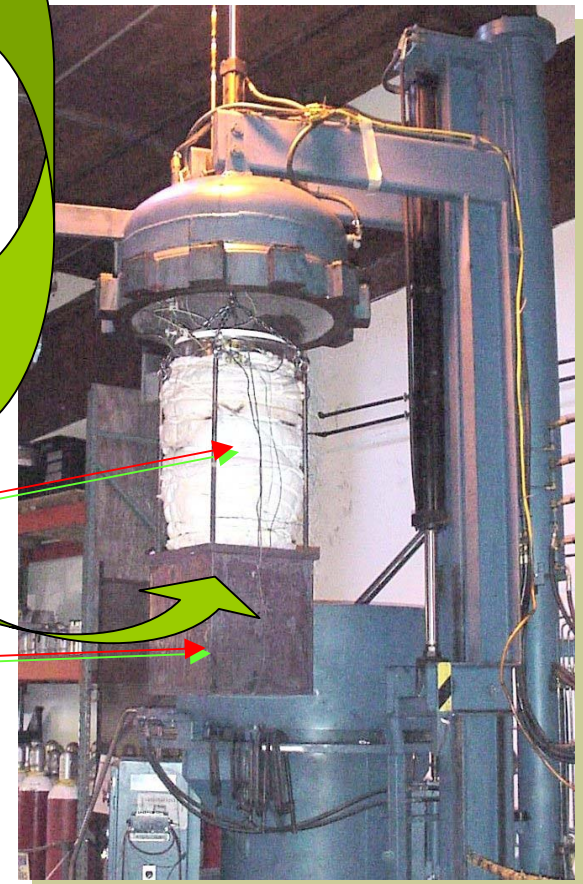
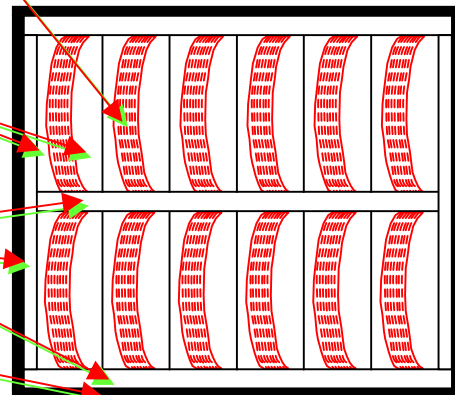
Graphite molds with figure machined cavity surfaces

Graphite mold constraints

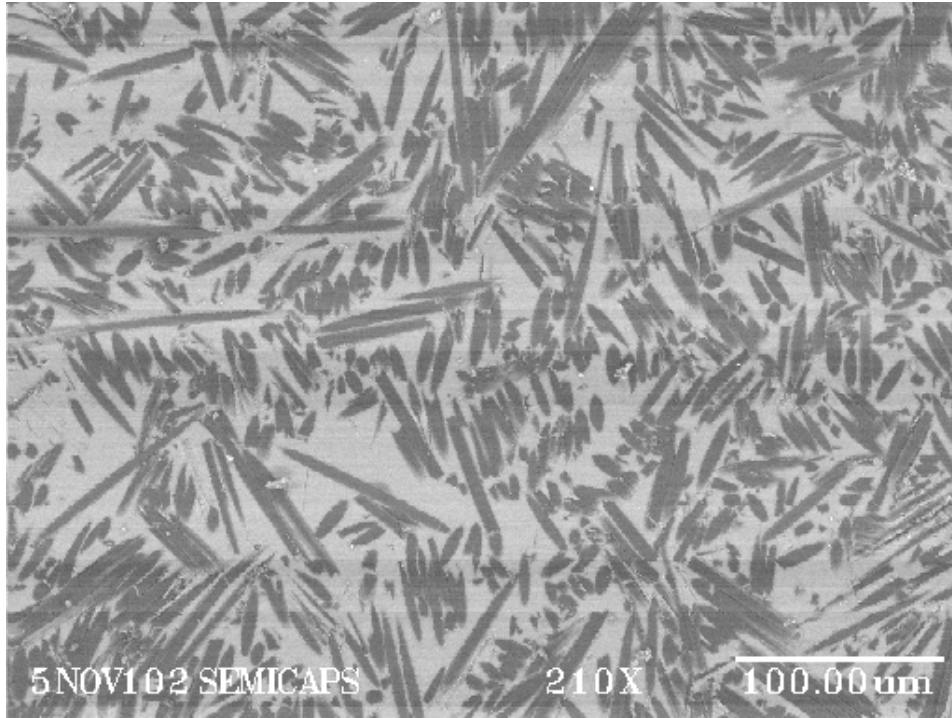
Vacuum tight steel vessel

Insulated molten alloy reservoir

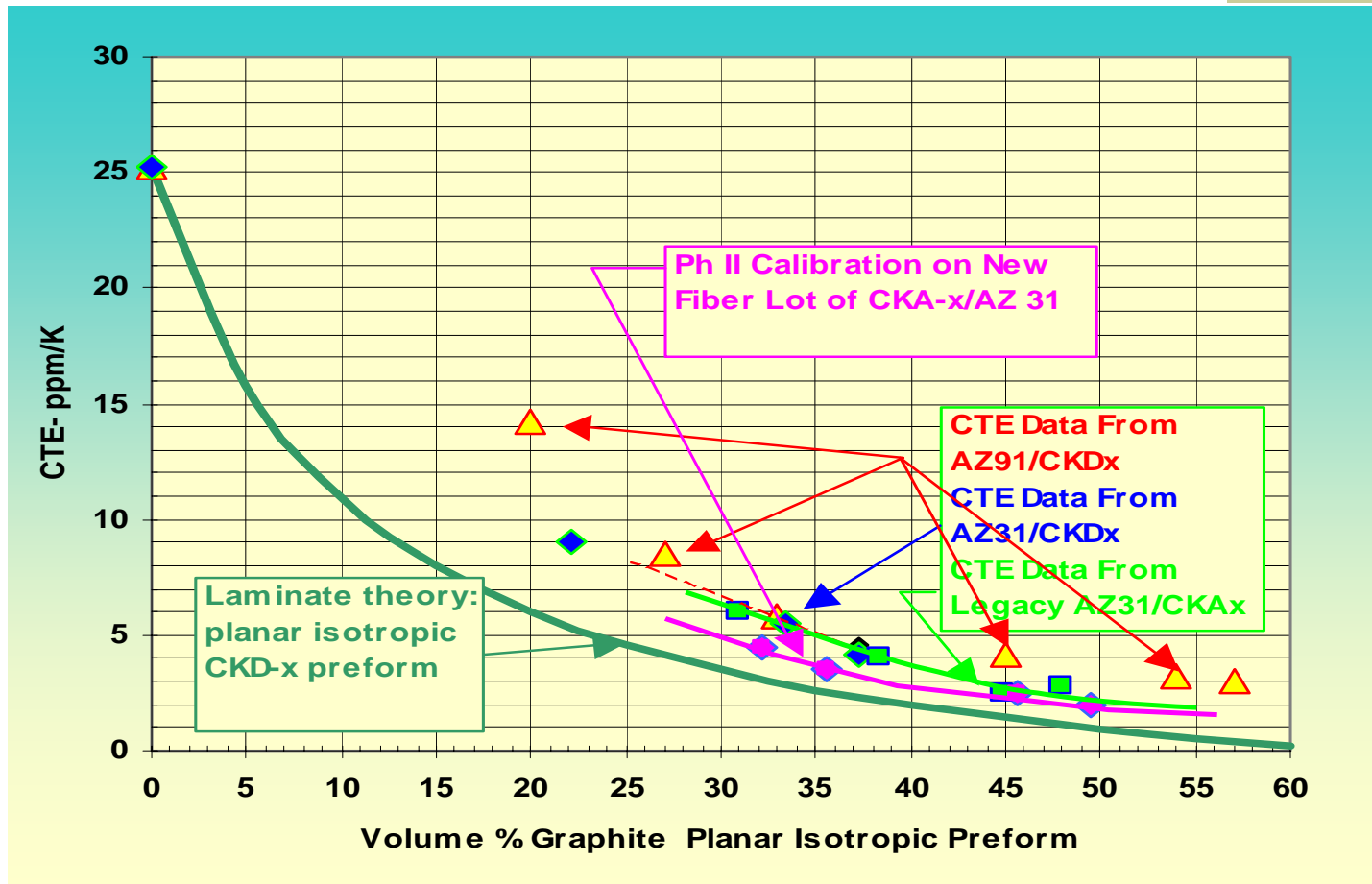
~2.5 Cubic Feet of preform material being transferred to autoclave for pressurization and liquid metal infiltration



In-Plane Microstructure of Gr/Mg Mirror Materials



Planar-Isotropic Mg/Graphite: Summary of CTE Data as a Function of v/o of various Gr Fiber preforms

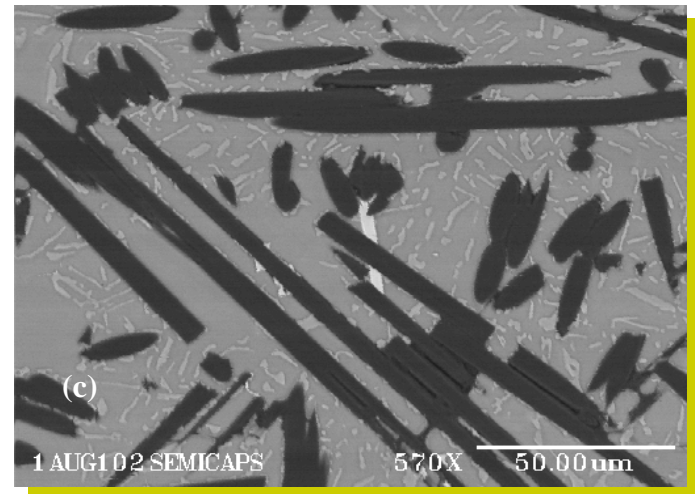
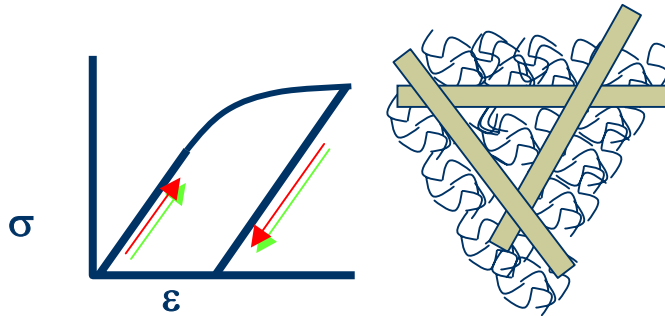
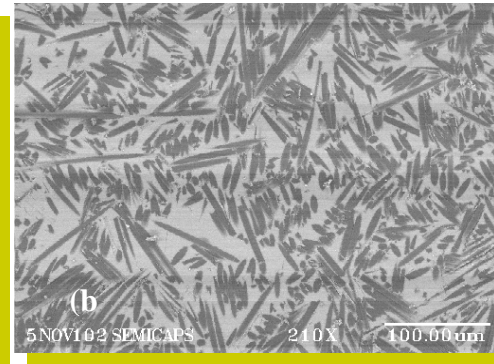
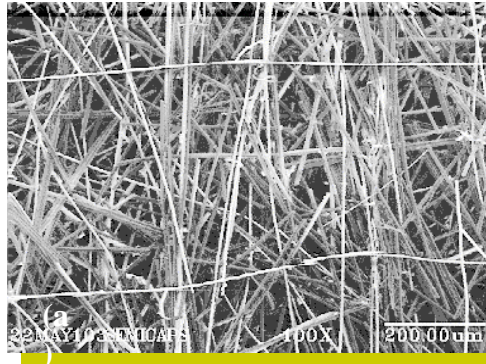


Expected Temperature Range of ABL

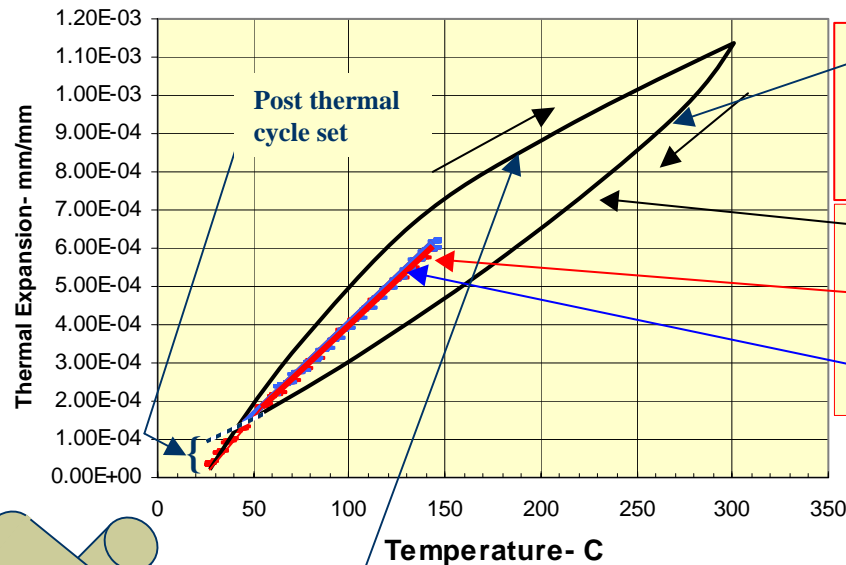
- **–20C to –65C in flight**
- **–15C to 46 C on the ground**

MMCC development stability goals:
50C to –70C

Thermal Stability Approach



Thermal Cycle of Mg AZ31 Reinforced with Planar Isotropic CKD-x Graphite Fiber

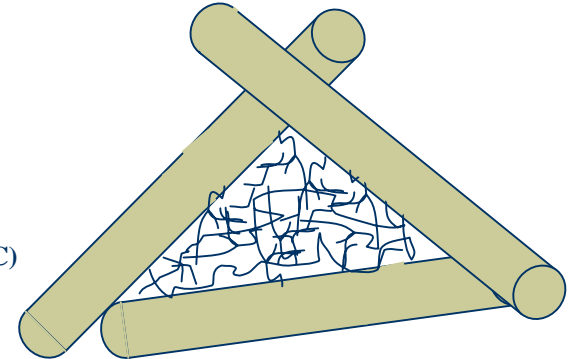
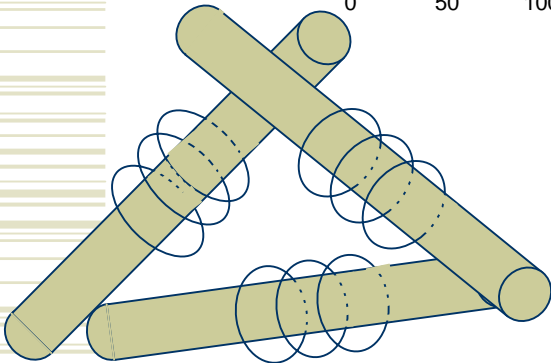


Cooling:

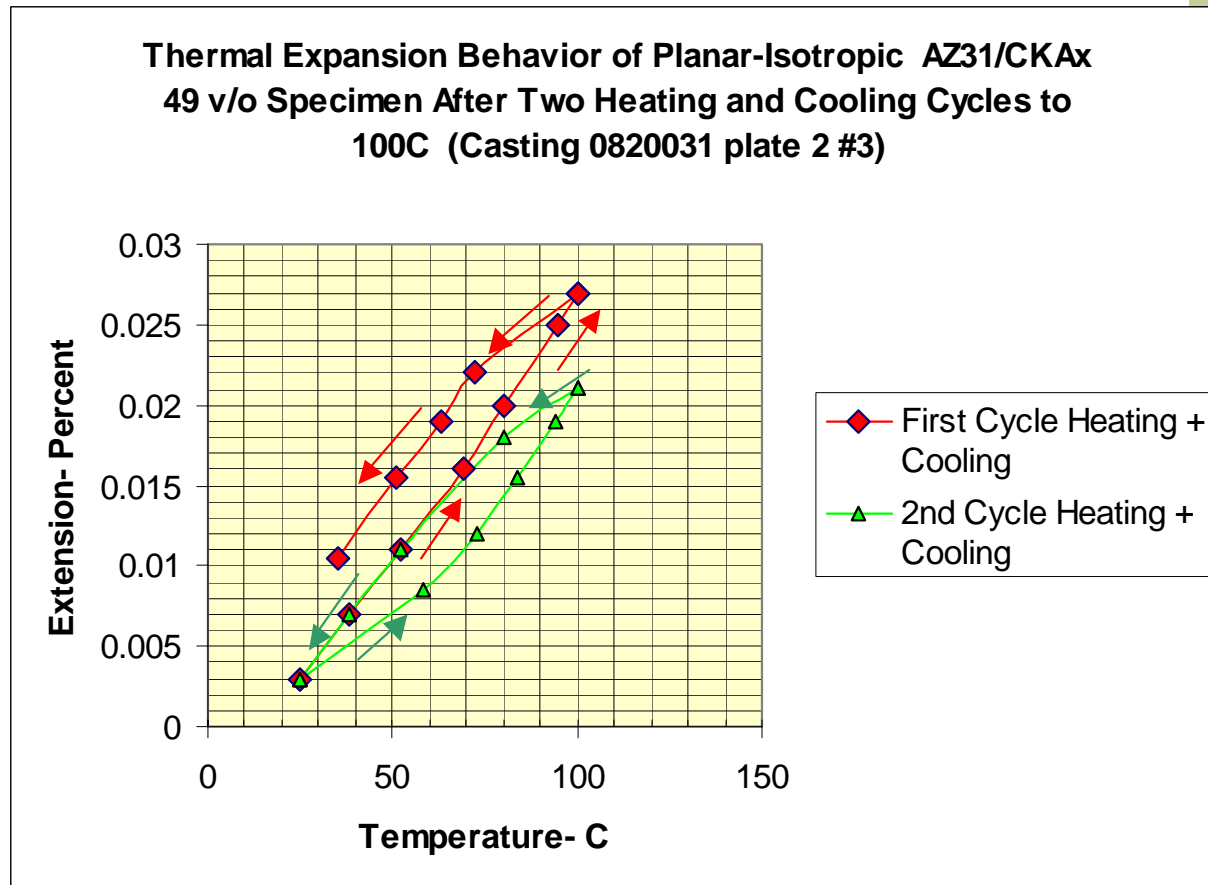
- Filament expands 1.5 ppm/K, Matrix shrinks 26 ppm/K
- Dislocations are punched to accommodate ΔCTE
- Dislocations are created faster than they are annihilated as temperature decreases below $0.5 T_m$, leading to a work hardened matrix
- Further thermal cycling is expected to rearrange dislocations into a stable substructure

Heating:

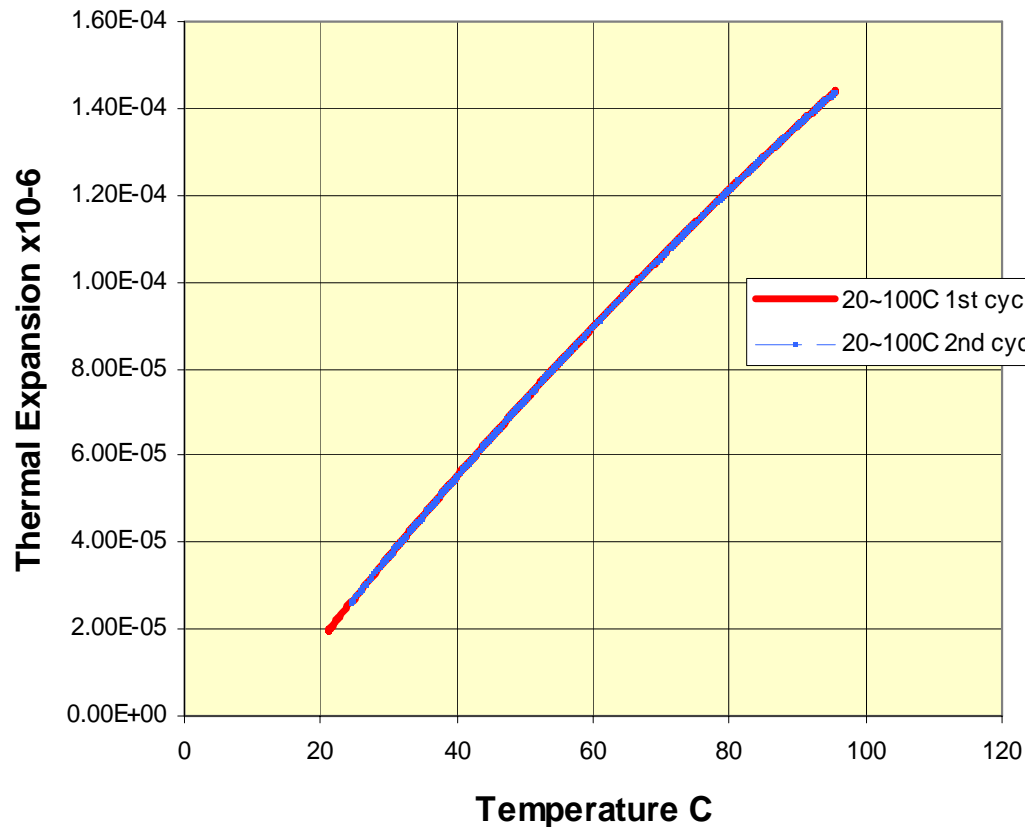
- Filament shrinks 1.5 ppm/K, matrix expands by 26 ppm/K
- Dislocations are punched into matrix to accommodate ΔCTE
- Dislocations are rearranged (or annihilated) as temperature approaches and exceeds $0.5 T_m$ (half melting point, 450K, 180C)



Example of Saturation upon Repeated Thermal Cycling, no Pre- Thermal Seasoning



Example of Complete Saturation and Stabilization after Pre-seasoning



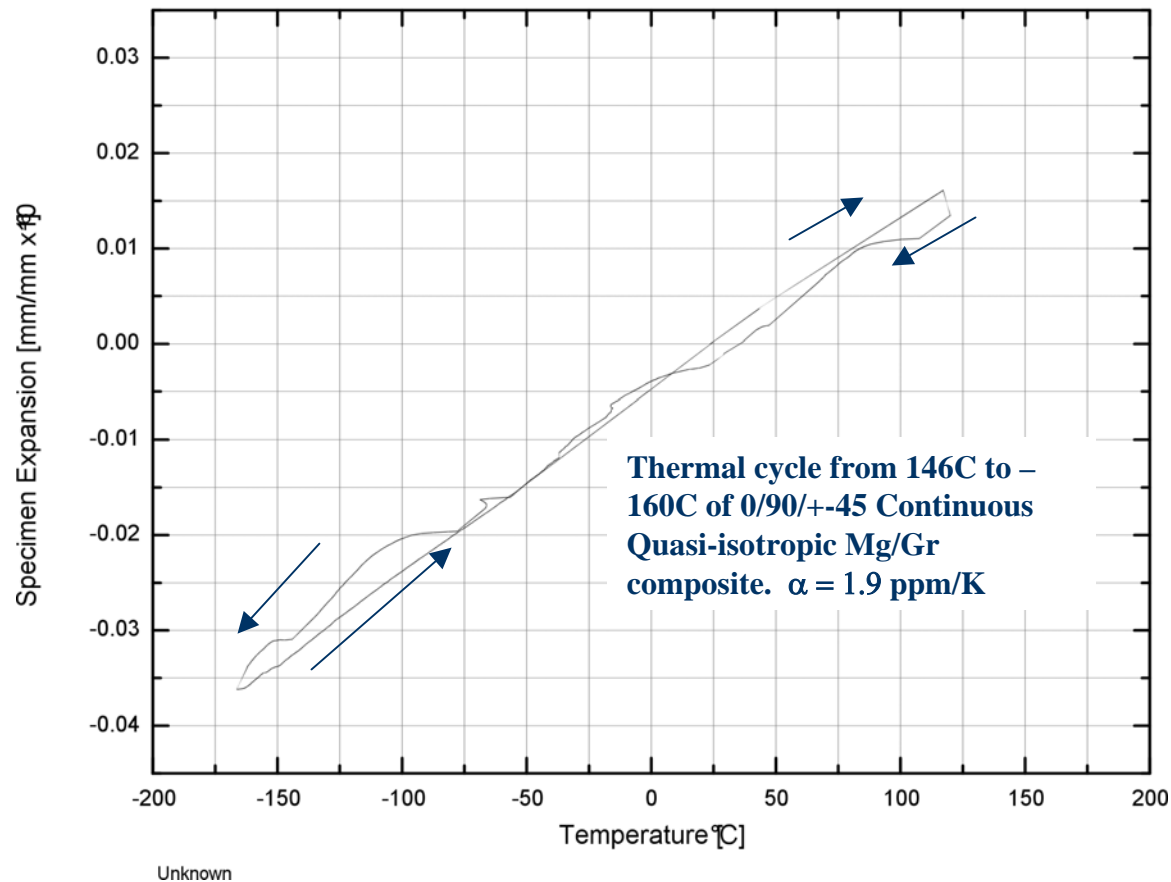
Seasoning HT:

**As cast + machined
plus two thermal cycles
from 25C to -75C.**

**Ave. CTE from 94C to
22C = 1.67 ppm/K**

Initial Cryogenic Thermal Cycle test

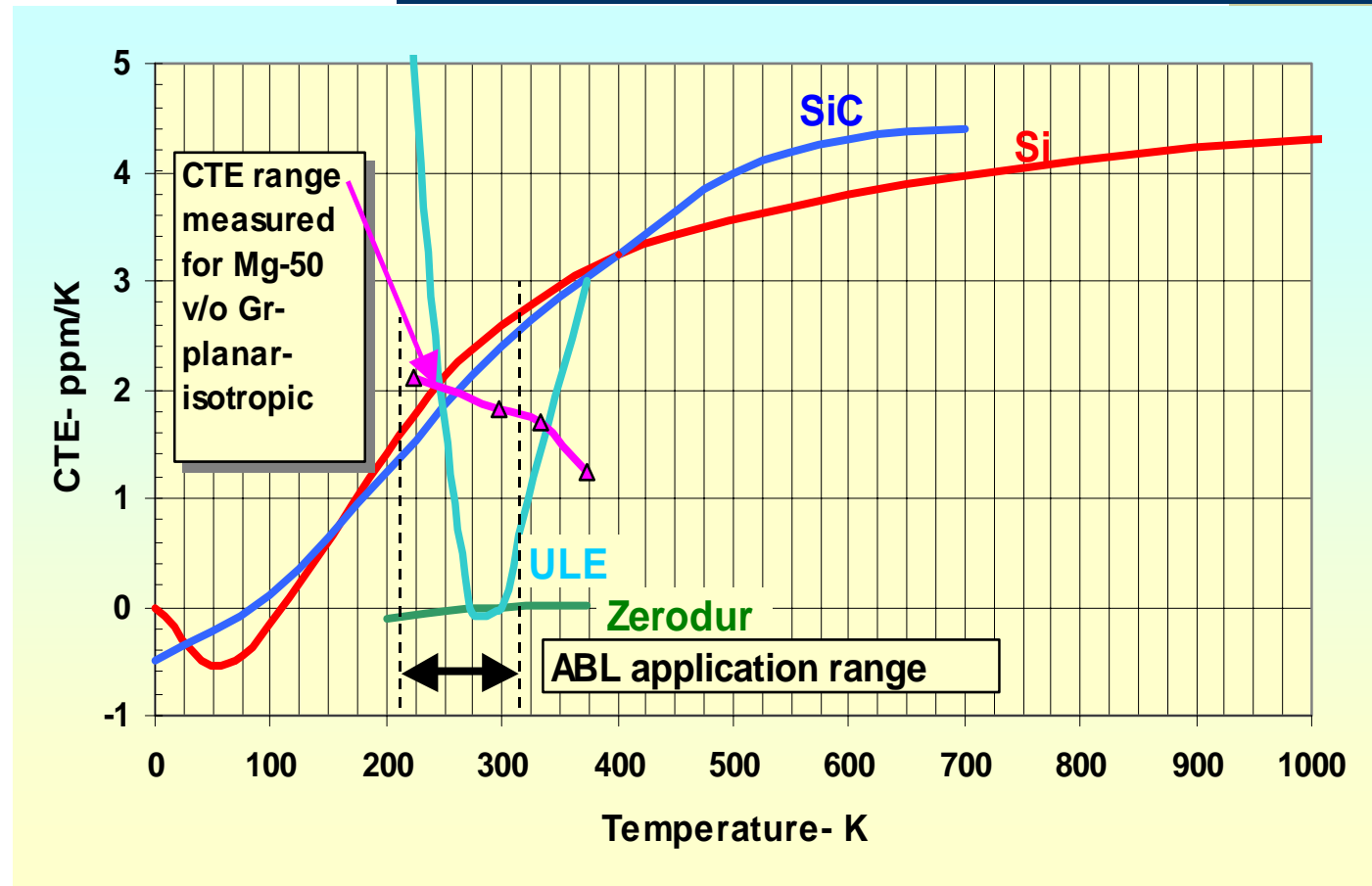
Specimen Expansion vs. Temperature



Temporary Manufacturing Specification for Gr/Mg ABL Mirrors

1. Preform to be CKA-x Paper Mat
2. Use proper ply rotation during preforming
3. Press to yield 50 v/o in the final casting
4. Pressure infiltrate with AZ 31 Mg alloy and directionally solidify
5. Machine to rough final shape in as-cast condition
6. Heat treat to 200C (TBD) for x min (TBD)
7. Slow cool to ambient
8. Thermal cycle from RT to 125C to -60C to RT to 125C to RT, n cycles to be determined.
9. Final machine to net shape
10. Post machining processing: (Electroplating, PACVD Si coating, replica attachment, etc.)

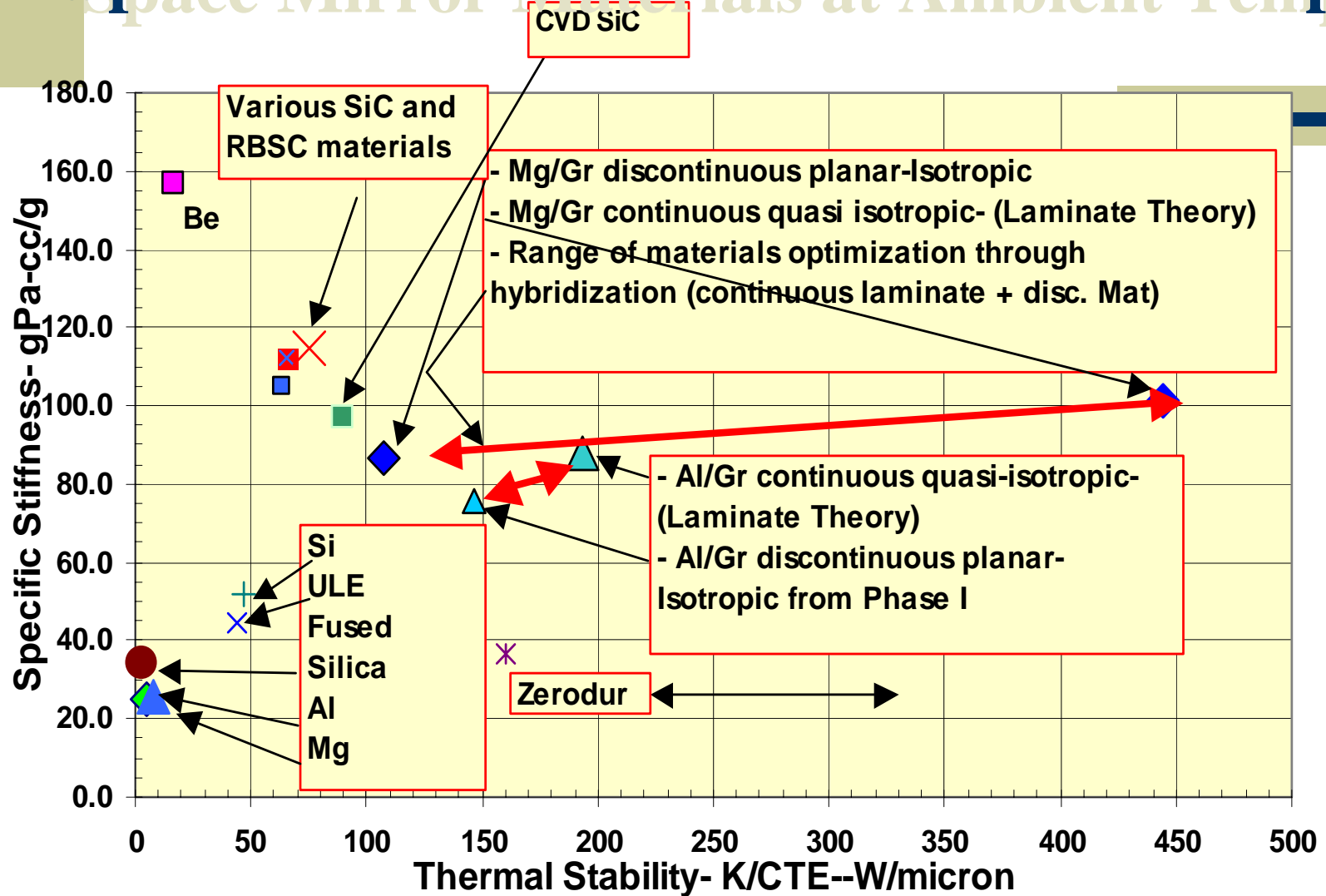
CTE of Mirror Materials (Si, SiC, ULE, Zerodur, Mg MetGraf) As a Function of Temperature



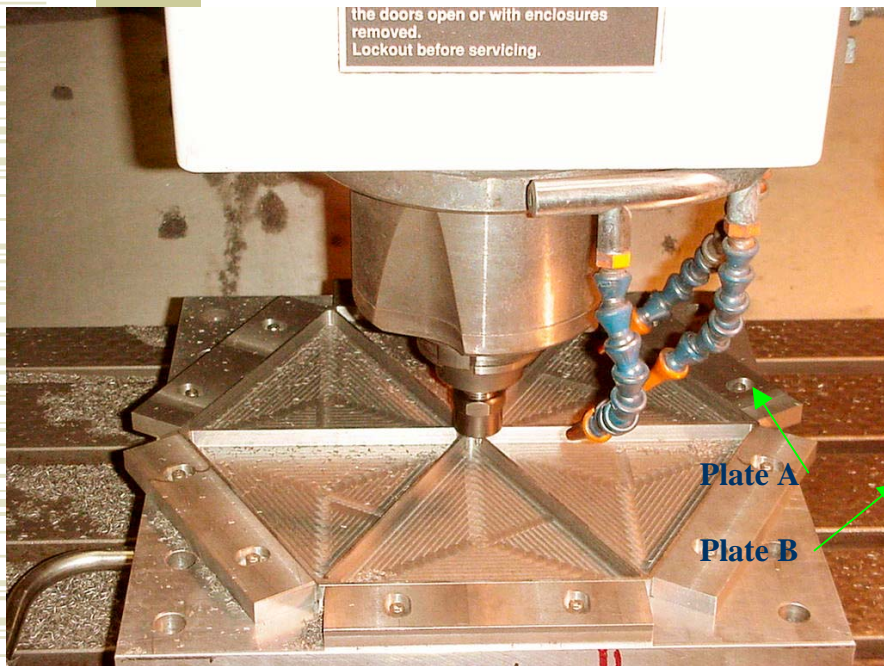
Properties of Candidate Space Mirror Materials

Material \ Property	Modulus E-gPa	density g/cc	Specific Stiffness gPa-cc/g	CTE ppm/K	Thermal Cond. W/mK	Thermal Stability K/CTE- W/mm	Reference
Mg	44.8	1.77	25.3	26	122	4.7	ASM Metals Hbk
Be	289.6	1.85	156.5	11.6	190	16.4	"
Al	69.0	2.70	25.5	22.7	170	7.5	"
ULE	98.0	2.21	44.3	0.03	1.31	43.7	Xinetics- NMSFC w orkshop 5/01
Zerodur	92.0	2.50	36.8	0.01	1.6	160.0	Poco- NMSFC w orkshop 5/01
Fused Silica	72.0	2.10	34.3	0.5	1.5	3.0	Poco- NMSFC w orkshop 5/01
Silicon	110.0	2.33	47.2	2.4	125	52.1	ASM Metals Hbk
Gr/Mg P-I Phase I	172.0	1.98	86.9	1.67	180	107.8	MMCC Ph. I
Gr/Al Phase I	180.0	2.38	75.6	1.5	220	146.7	MMCC Ph. I
Ceraform SiC	310.0	2.95	105.1	2.44	156	63.9	Xinetics- NMSFC w orkshop 5/01
Poco SiC	248.0	2.55	97.3	1.9	170	89.5	Poco- NMSFC w orkshop 5/01
CVD SiC	448.0	3.21	139.6	2.6	240	92.3	
SSG HP SiC			112.0			65.0	SSG- NMSFC w orkshop 5/01
SSG RB SiC			115.0			75.0	"
Mg/Gr Quasi Isotropic	203.0	2.00	101.6	0.45	200	444.4	JC Lam. Theory + ROM for K
Gr/Al Quasi isotropic	210.0	2.37	88.6	1.14	220	193.0	"

Comparison of Key Properties of Candidate Space Mirror Materials at Ambient Temp.



Demonstration of Lightweighting in Planar Isotropic Al/Gr Composites



Total machining time=3 hr

Plate 1: Face thickness=2 mm. Outer ribs = 1.75 mm, Inner ribs = 2 mm, Mass in Al/Gr = 375 g, Areal density in Al/Gr = 6.12 kg/m² Equivalent Areal density in Mg/Gr = 5.04 kg/m²

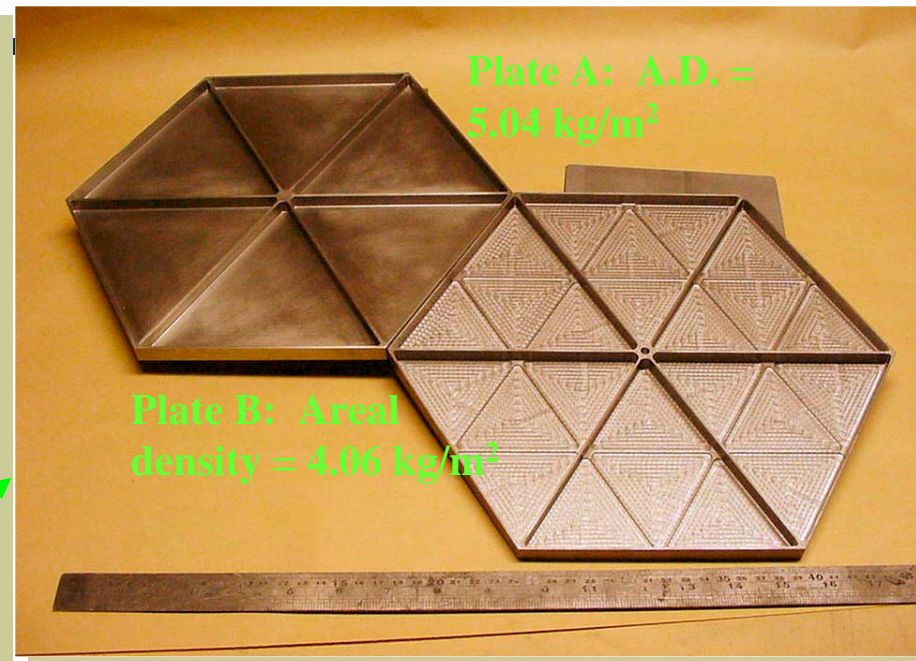
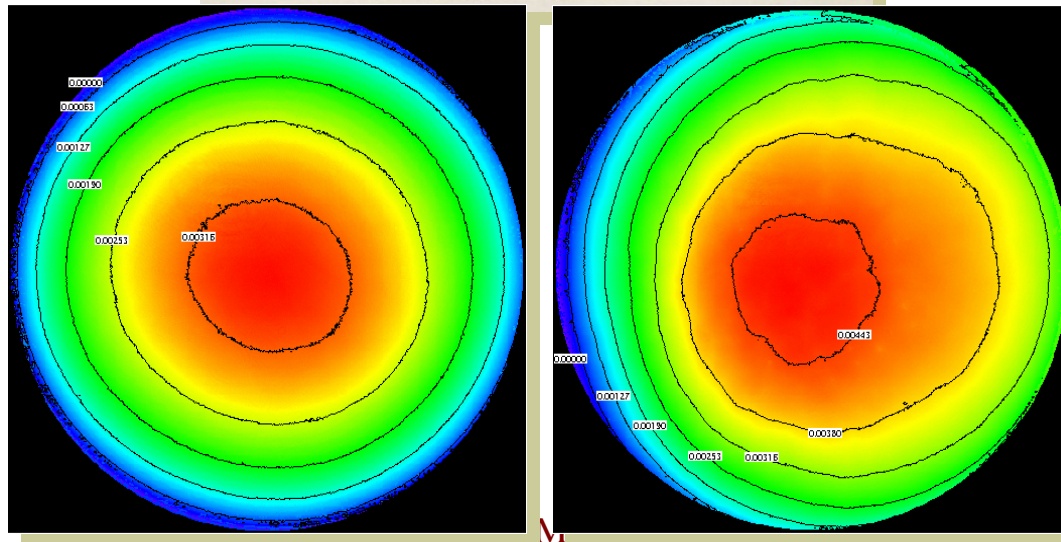


Plate 2: Face thickness = 1.625 mm, Outer ribs = 1.5 mm, Inner ribs = 1.375 mm, sub-ribs 1 mm wide x 1 mm deep. Mass in Al/Gr = 307 g, Areal density in Al/Gr = 5.04 kg/m², equivalent areal density in Mg/Gr = 4.06 kg/m²

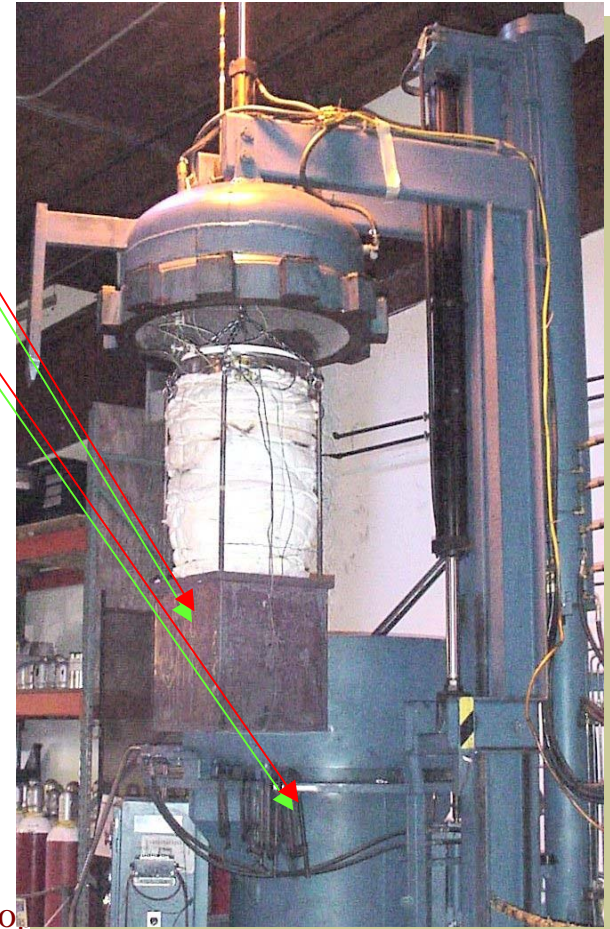
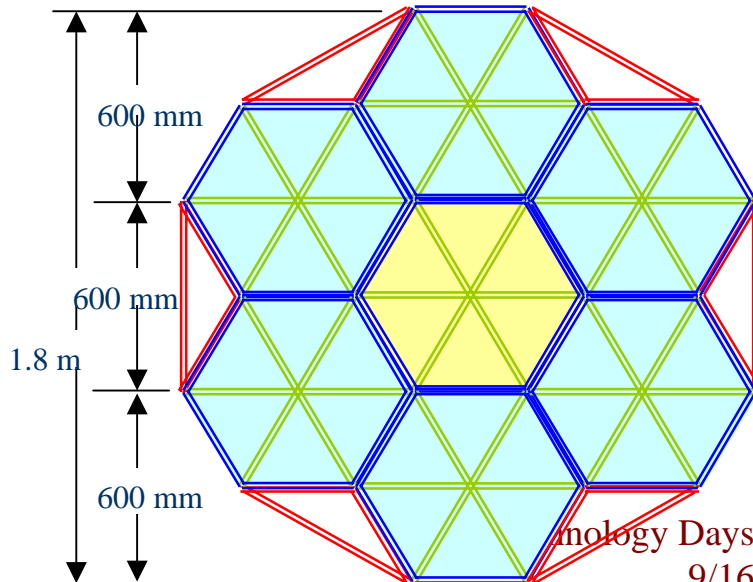
Si Coated and Polished Gr/Mg and Gr/Al



MMCC's Manufacturing Scale Pressure Infiltration Casting Facility

3 1/2 Cubic Feet of preform material being transferred to autoclave for pressurization

30" diameter autoclave which will permit ~ 4-7 60 cm diameter mirror segments per casting. An 1.8 m mirror can be assembled from 7 hex segments + six filler segments.



Technology Days: Mirror Development
9/16-9/18, '03

Summary of MMCC Mirror Manufacturing Process

1. Stack planar-isotropic (random in-plane) mats or quasi-isotropic lamina in hydraulic press mold shaped to near final figure
2. Compress to desired volume fraction and set binder
3. Insert preform into pre-figured mold cavity
4. Heat under vacuum to evaporate binder
5. Pressure infiltrate with molten Mg (or Al) and solidify
6. Lightweight machine back plane, machine mirror plane to near-final figure to a roughness less than $10\ \mu$
7. Plasma CVD coat with $\sim 125\ \mu$ Si
8. Polish to final figure and finish

Conclusions from AF Phase I Mg/Gr Mirror Study

- Calibration curves for CTE developed. Projected goal of 2 ppm/K and >150 W/mK thermal conductivity met and exceeded with ~55 v/o CKA-x isotropic preform. Actual CTE measured was 1.96 ppm/K and thermal conductivity was 160 W/MK. (Recalibrated to 50 v/o for modified Phase II preform)
- An areal density of 5.01 kg/m² was demonstrated in Al/Gr. This converts to 4.02 kg/m² for Mg/Gr composites. Further mass reduction is possible.
- Planar isotropic CKA-x preforms resulted in reliable CTE properties.
- CTE behavior of Al-12.5 Si-.3 Mg matrix reinforced with planar isotropic CKD-x fiber matches laminate theory and results in extremely well behaved materials with high thermal conductivity.
- With current facilities, 610 mm segments can be manufactured. These can be assembled into 1.5 to 1.8 m or greater mirrors.

Conclusions from AF Phase I Gr/Mg Mirror Study- (continued)

- A larger autoclave (48" dia) will enable manufacture of 1000 mm hex segments (*autoclave cost est. @ \$160k*)
- Hybridized planar-isotropic mats/quasi-isotropic angle ply continuous fiber laminate Gr/Mg can be tailored to CTE of 0 ppm/K
- ♦ CTE does not change with temperature from ambient to 140C. Indications are that CTE will be constant to ~ -60C
- ♦ Thermal seasoning to -75C produces thermal stability over ABL thermal exposure range (46C to -65C)
- ♦ Material is extremely easy to machine. Since Si plating is needed for mirror surface, figure and finish during preparation of substrate is not critical

Future R&D Required

- Optimization of Mg/Gr preforms by perfecting starting mat material and through hybridization with continuous angle-ply laminates
- Optimize matrix alloy for high thermal conductivity
- Develop/demonstrate limits of thermal stability for various applications:
 - Airborne (**Preliminarily demonstrated**)
 - Near and deep space
 - Ambient (**Demonstrated**)
- Develop matching CTE for metering structures and optical benches for
 - Gr/Mg
 - RBSiC
 - ULE, Zerodur, Be, Invar, etc

Phase II Technical Objectives

Overall Phase II technical objectives are:

1. Optimize graphite fiber reinforced Mg alloy systems for mirrors and mirror substructures (optical benches, metering structures)
2. Demonstrate that the MMCC technology can be used to fabricate 500 mm hexagonal mirror segments
3. Demonstrate that the hex mirror segments can be joined to form a 1500 mm mirror.
4. Demonstrate that the mirror structure in the finished form will weigh less than 19.3 kg/m^2 . (4 kg/m^2 was demonstrated in Phase I on an arbitrary design)
5. Demonstrate that the mirror substructure can be Si coated and polished to an aspheric figure with the following specifications:
 - PV better than 1.0 waves
 - RMS better than 0.25 waves
 - Roughness better than 10 nm rms

Phase II Technical Objectives (continued)

More specific technical objectives include the following:

6. Optimize a Gr/Mg alloy system for maximum thermal cycling stability over ABL temperature range
7. Optimize the composite system for a CTE of ~ 2 ppm/K with high conductivity and high specific stiffness
8. Demonstrate potential for lower, e.g. 1 to 0 ppm/K CTE
9. Demonstrate dimensional stability during thermal cycling
10. Develop, in parallel, the materials science necessary to understand and predict the materials behavior
11. Explore potential for organic replicas applied to Mg/Gr flats

Non technical objectives are:

1. Demonstrate that after the prototype and scale up development is completed, that the procurement time for a mirror is less than 18 months
2. Demonstrate that the mirror is cost effective compared to Be and ULE

New Phase I MDA Mirror Projects

- ♦ **MDA-03-025** (B031-0921)
“Light Weight Thermally Balanced Graphite Reinforced Mg Structural Substrates for Replicated Mirror Membranes”
- ♦ **MDA-03-048** (B031-0925)
“Graphite Fiber Reinforced Magnesium as a Beryllium Replacement Material for EKV Seeker Mirrors and Substructures”

MDA-03-025 Objectives:

Replicated Mirror Membranes on Mg/Gr Substrates

1. Determine feasibility for developing inexpensive and light weight mirrors with short delivery times using MMCC's Mg/Gr technology and best practice replica technology by CRG and LLNL
2. Demonstrate that MMCC's Mg/Gr technology can be scaled to large segmented mirrors up to 10 m diameter
3. Demonstrate that neat cyanate ester (NCE) replicas can be transferred to a Mg/Gr substrate and maintain float-glass optically flat figure and micro-finish of the master optical mandrel
4. Demonstrate that the LLNL Cu-Zr (or Ni-Ti) nanolaminates can be transferred to a Mg/Gr substrate and maintain the optically flat figure and micro-finish of the master optical mandrel
5. Demonstrate a optical surface sheet stiffness of ~200 gPa and CTE matching to the nanolaminate

MDA-03-025 Objectives: Replicated Mirror Membranes on Mg/Gr Substrates- (continued)

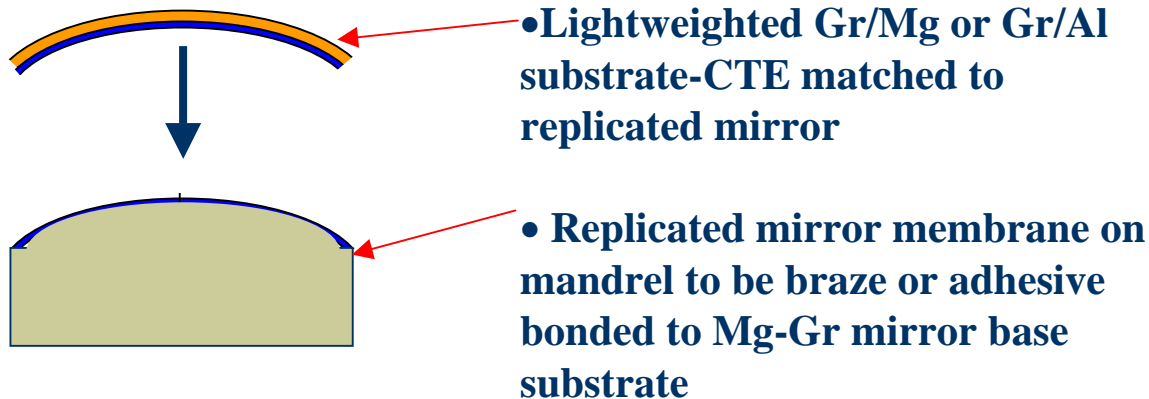
- 6. Demonstrate that a mirror structural substrate can be manufactured without knit patterns and print-through in the final replicated surface**
- 7. Demonstrate rigid mirror structural substrate areal density less than 10 kg/m²**
- 8. Demonstrate that a symmetrical and structurally stable structural substrates can be reliably manufactured and demonstrated with replicas of flat float-glass master mandrel attached**

Specific questions to be answered from the Phase I research are:

1. Can the proposed surface architecture with discontinuous fibers arranged randomly in-plane prevent knitting and print-through of angle ply asperities in fiber distribution?
2. Can the surface figure be maintained after thermal cycling over the application temperature range?
3. Can the NCE replica be extracted from the master optical mandrel without stretch marks and distortions?
4. Can CTE be optimized to the replicated membrane and still maintain attractive stiffness?
5. Can the replica be applied to the structural substrate without unbalancing the structure?
6. Can the structural substrates be manufactured economically and with rapid delivery?
7. Which replica technology (CRG polymeric or LLNL nanolaminate Ti-Ni (or others such as TREX SiC membranes)) offers most promise when combined with MMCC's Mg/Gr structural substrates?

Alternative Approach to Lightweight Mirrors:

Rigid lightweighted mirror base with replicated surface film, adhesive bonded

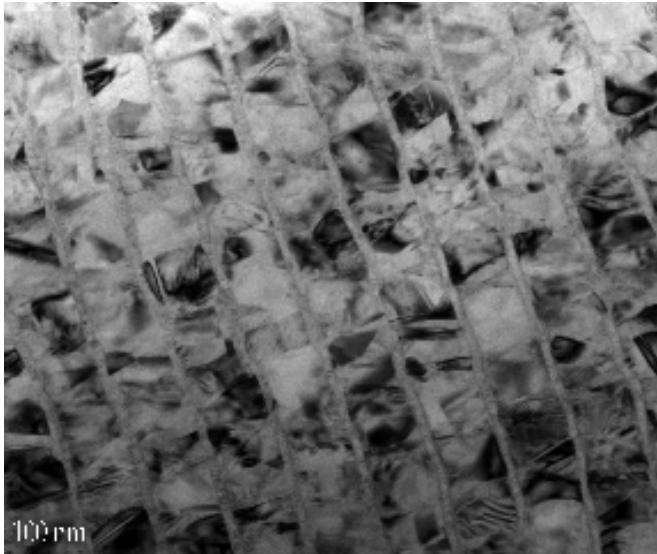


Neat Cyanate Ester Replica

**CRC's cyanate ester-glass
syntactic composite
mirror**



Nanolaminate Replica Membranes



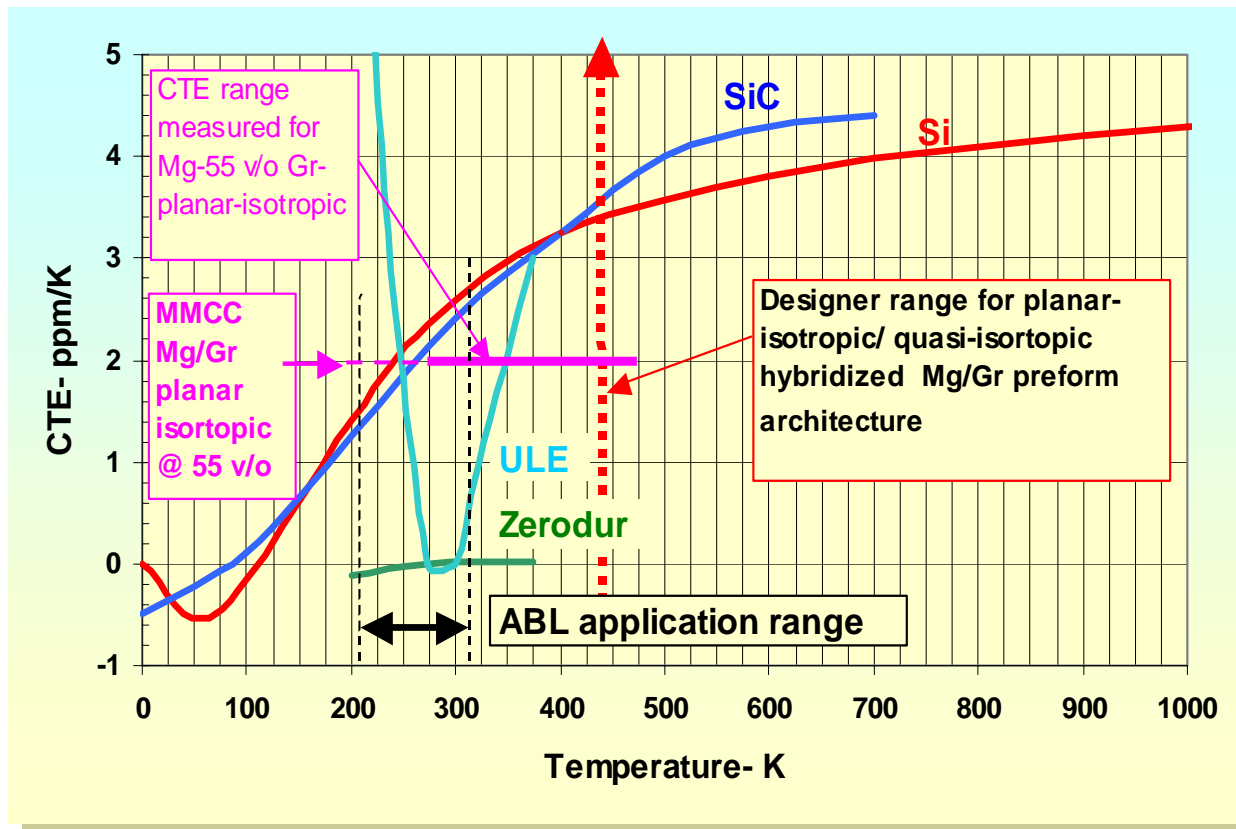
A 25 cm diameter, 110 mm thick thin shell mirror consisting of alternating 600 Å thick copper layers and 80 Å thick copper/zirconium amorphous intermetallic layers is shown. A surface finish of 10 Å achieved off a super-polished tool.

(Compliments of Dr. Troy Barbee)



Technology Days: Mirror Development
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CTE Compatibility of Candidate Mirror Materials with Mg/Gr



MDA-03-025

Work Plan

2.1 Materials evaluation and preliminary optimization for replica substrate

- 2.1.1 Quasi-isotropic architecture (0, +/-60)
- 2.1.2 Hybridized (next generation mat + quasi-isotropic)
- 2.1.3 Legacy architecture using planar-isotropic mats @ 55 v/o

2.2 Materials evaluation

- CTE, E, TS, TC,
- Thermal seasoning
 - ♦ Dimensional stability
 - ♦ Flatness

2.3 Materials selection and fabrication for replication studies

Work Plan (continued)

2.4 Manufacture materials for CRG application of Neat Cyanate Ester (NCE) replicas and LLNL microlaminates

- ♦ ~24- 32mm x 32 mm x 10 mm thick blanks polished to ~10 μ roughness
- ♦ ~18- 75 mm diameter x 10 mm polished to ~10 μ roughness
- ♦ 12 hex flats- 150 mm x 10 mm thick lightweight machined to 10-15 kg/m², polished to ~10 μ
- ♦ All blanks to be thermal seasoned prior to replica application

3.0 Replica application

3.1 NCE applied to MMCC substrates at CRG

3.2 Microlaminates applied at MMCC with consultation from LLNL

3.3 Evaluation:

- ♦ CRG to perform test matrix using NCE and evaluate finish, PV
- ♦ MMCC to evaluate thermal cycling from – 100 to + 100C.
Dr. Mollenhauer AFRL/MLLM to evaluate PV and finish

MDA-03-025

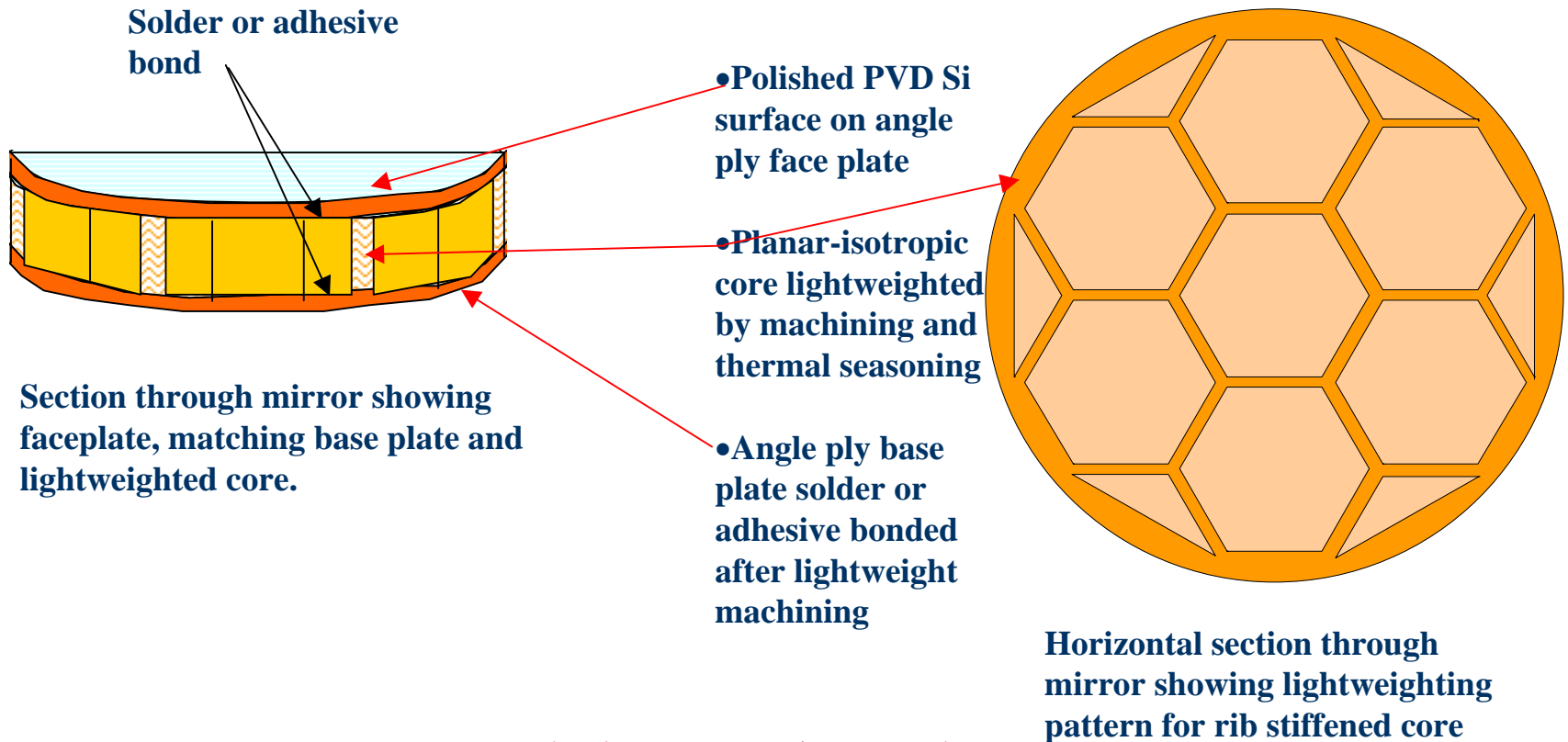
Work Plan (continued)

4.0 Demonstrate feasibility using ABA as a focus and reference model for the study

- ◆ **Cost analysis**
- ◆ **Buy to fly time line analysis**
- ◆ **Figure and finish fidelity of replicated optics**
- ◆ **Print through analysis after thermal cycling**

MDA-03-048

“Be Buster”(B031-0925)



MDA-03-048

Overall Objectives (B031-0925)

- 1. Demonstrate that stable Gr-Mg structures can be manufactured**
- 2. Demonstrate thermal stability**
- 3. Demonstrate technology by manufacturing a mirror structure with mass and performance equivalent to Be for EKV primary steering mirror**
- 4. Demonstrate manufacturing cost savings of Mg-Gr composite mirrors compared to Beryllium**
- 5. Additional post-award objective by PI: Can thin film, e.g. SiC improve performance of Gr/Mg substrate mirror?**

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Overall Objectives (B031-0925)

(continued)

The principle technical question to be answered is:

Can thermal seasoning eliminate hysteresis during thermal cycling and result in thermal stability?

Gr Fiber Reinforced Mg as a Be Replacement Material for EKV Seeker Mirrors Phase I (F29601-03-M-0280)

Additional suggestions by Prime KEV contractor:

- **In MMCC design, CTE of Mg/Gr should match CTE of Si at 300K (RT) (~2.6 ppm/K). This corresponds to decreasing the planar isotropic volume fraction CKAx fiber from 50 (for ABL applications) to 42-45 v/o.**
- **Thermal stability to be based on #A10FC-1 (Coastal Alaska) maximum diurnal temperature variation of -35 to 44C (less severe than ABL).**
- **Fundamental vibration frequency to be 2500 Hz.**
- **Demonstrate technology with f-1 (based on 8" aperture) with a RC of 16".**